

BT15 Rec'd PCT/PTO 03 JAN 2005

1

DESCRIPTION**FIBER PROCESSING AGENT AND FIBER PROCESSED WITH THE FIBER
PROCESSING AGENT****5 Technical Field**

The present invention relates to a fiber processing agent and fiber processed with the fiber processing agent.

Background Art

10 There have been various products using fiber such as blouse, dress shirts, pants, skirts, backing clothes, covering materials for furniture and seats of vehicles and the like in the market.

The fiber as a material for producing the products is required to have various characteristics for satisfying needs in each field of application. The required
15 characteristics include, for instance, moisture retention, water-absorbing property, moisture-absorbing property, antistatic property.

For instance, blouse is worn casually, and a wearer often gets sweaty while wearing the blouse. Therefore, the moisture-absorbing property is required to the blouse. There are various types of fibers capable of satisfying the requirement for
20 moisture-absorbing property. The fibers as described above include, but not limited to: synthetic fiber such as nylon, polyester, acryl and polyurethane; natural fibers such as cotton, linen, wool; and compound fibers prepared with the synthetic and natural fibers.

Sometimes the fibers are subjected to processing with a specific fiber processing agent for improving the properties described above, or for adding any other specific
25 property other than those described above.

For instance, the eggshell membrane has, in addition to the capability of improving the properties described above, the wound-treating property of promoting cure of a wound when applied to a surface of wounded skin, and there has been known the use of the eggshell membrane as a sheet material prepared by mixing powder of eggshell

membrane in a fiber material and forming the mixture into a thin sheet so that the sheet material can easily be applied to a surface of wounded skin.

In order to improve the wound-treating property of the sheet material made of a fiber material having the wound-treating property as described above, there has been
5 proposed a method of preparing an aqueous solution with the eggshell dissolved therein and immersing woven cloth or unwoven cloth in this aqueous solution and then drying the cloth to produce a sheet material (Refer to Japanese Patent Laid-Open Publication No. HEI 7-246234).

So long as the sheet material prepared by the method is used only once as a
10 disposable material, the characteristics such as the wound-treating property is provided, and thus there occurs no problem.

With the technology disclosed in the reference described above, however, as woven cloth or unwoven cloth is immersed in an aqueous solution containing only eggshell membrane and then the cloth is dried, adhesion of the eggshell membrane to the
15 sheet material is weak. Therefore, for instance, when the sheet material is washed repeatedly or used for a long period of time, the eggshell membrane is removed with the durability lowered, and such properties as moisture retention property, water-absorbing property, moisture-absorbing property, antistatic property, and wound-treating property can not disadvantageously be retained for a long time.

20

Disclosure of the Invention

An object of the present invention is to provide a fiber processing agent having excellent durability and capable of continuously providing moisture retention property, water-absorbing property, moisture-absorbing property, antistatic property and
25 wound-treating property, as well as fibers processed with this fiber processing agent.

The fiber processing agent according to the present invention is used for processing a surface of fiber, and contains soluble eggshell membrane and a reactive organic compound having a reactive group.

In the present invention, the soluble eggshell membrane indicates an eggshell

membrane of a bird's egg (represented by a hen's egg) which is chemically- processed with an agent such as an acid, an alkali, an oxidizing agent, or a reducing agent into the water-soluble state.

In addition to the hen's egg, eggs of goose, quail, ostrich, and the like may be
5 used as materials for the eggshell membrane according to the present invention.

Representative reducing agents which may be used in the chemical processing according to the preset invention include thioglycolic acid, thiopropionic acid, and 2-melcaptoethanole.

For preparing the soluble eggshell membrane used for production of a fiber
10 processing agent, at first, an aqueous solution of the soluble eggshell membrane is prepared, and then any method may be used for preparing the fiber processing agent. In one of the representative preparation methods, 10 to 32 weight portions of the aqueous solution of thioglycolic acid (14.3 mol with the concentration of 100% in the liquid phase) or thiopropionic acid (11.3 mol with the concentration of 100% in the liquid phase), or a
15 mixture thereof with the concentration of 1 mol/litter or more is added to one weight portion of the eggshell membrane.

Then the mixture solution is heated to a temperature in the range from 50 to 70 °C, and is kept under the temperature for five to several tens hours until the eggshell membrane is completely dissolved. Then the reducing agent used in the reaction is
20 removed to obtain an aqueous solution.

More specifically, acetone is added to the solution above to precipitate the eggshell membrane having been processed into the soluble state and dissolved therein, the solution (reducing agent) is removed, and then the precipitate is washed with acetone once or twice, and then water is added to the precipitate to obtain an aqueous solution.

25 Alternatively, the reducing agent solution with the eggshell membrane dissolved therein is processed into the acidic state with hydrochloric acid for preventing oxidization, and then the solution is dialyzed through water to remove the reducing agent.

When 2-melcaptoethanole is used as a reducing agent, the pH is adjusted to the alkaline side in the range from about 9 to 10 for strengthening the reducing capability, and

is heated for five to several tens hours at a temperature in the range from 50 to 70 °C. In this case, as it is difficult to completely dissolve the eggshell membrane, the processing is terminated within a prespecified period of time, and then impurities are removed by centrifugation, and the remaining solution is dialyzed through water, or the dissolved eggshell membrane is precipitated with acetone, and the precipitated eggshell membrane is dissolved, for instance, in water to obtain an aqueous solution of soluble eggshell membrane.

Further, with the soluble eggshell membrane is prepared by dissolving the eggshell membrane with alkali, an aqueous solution of 1 to N (specified value) sodium hydroxide or that further containing alcohol (with the alcohol density of 50 to 70%) is added to one weight portion of eggshell membrane, the mixture solution is heated for 3 to 6 hours at a temperature in the range from 40 to 60°C, and then is dialyzed through water for neutralization.

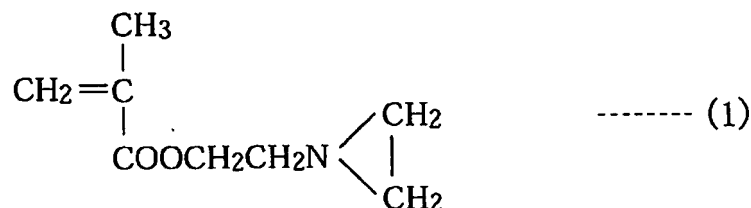
In the fiber processing agent according to the present invention, the reactive organic agent preferably contains one or more selected from the group consisting of (component 1) a hydrophilic compound having a polymerizable vinyl group in the molecule, (component 2) a monomer containing any of a hydroxyl group, a carboxylic group, an amino group, a sulfonic group, and a phosphate group, (component 3) a hydrophilic compound having an epoxy group, and (component 4) a compound having an aziridine group.

Specifically, the (component 1) above is any of polyethylene glycol diacrylate, polyethylene glycol dimethacrylate, bisphenol A polyethylene glycol diacrylate, bisphenol A polyethylene glycol dimethacrylate, bisphenol S polyethylene glycol dimethacrylate, and the like.

Specifically, the (component 2) is any of acrylic acid, methacrylic acid, maleic acid, itaconic acid, acrylamide, methacrylamide, vinyl sulfonic acid, hydroxylpropyl methacrylate, and the like.

Specifically, the (component 3) is polyethylene glycoldiglycidyl ether or the like.

Specifically, the (component 4) is, for instance, a compound having the following formula:



5 According to the present invention, the aqueous solution of soluble eggshell membrane described above and the reactive organic compound are mixed with each other to prepare the fiber processing agent.

In addition to the soluble eggshell membrane and the reactive organic compound, such materials as fibroin, sericin, and chitosan may be blended therein. When the
10 material or materials are blended therein, the moisture-absorbing property is improved.

With the present invention as described above, because a reactive organic compound having a reactive group is contained therein, even when the fiber processed with the agent is used for a long period of time, the eggshell membrane is never removed. Therefore, the various properties of the eggshell membrane are maintained, so that a fiber
15 processing agent with excellent durability and also continuously providing the moisture retention property, water-absorbing property, moisture-absorbing property, antistatic property, and wound-treating property can be obtained.

In the fiber processing agent according to the present invention, preferably the soluble eggshell membrane is contained in the range from 0.1 to 10% by weight relative to
20 the total weight of the fiber processing agent and the reactive organic compound in the range from 1 to 20% by weight. More preferably, the soluble eggshell membrane is contained in the range from 0.2 to 5% by weight and the reactive organic compound in the range from 2 to 10% by weight.

When the concentration of the soluble eggshell membrane is less than 0.1% by
25 weight and that of the reactive organic compound is less than 1% by weight, effects of the moisture retention property and the like are insufficient. When the concentration of the

soluble eggshell membrane is more than 10% by weight and that of the reactive organic compound is more than 20% by weight, the fiber processed with the agent may become stiff.

- 5 The fiber processing agent according to the present invention may have also the composition as described below.

 The fiber processing agent according to the present invention is used for processing a surface of fiber, and contains a soluble eggshell membrane and an organic compound having the adhesiveness.

- 10 The soluble eggshell membrane is described as that contained in the fiber processing agent according to the present invention above, so that description thereof is omitted herefrom.

- In the fiber processing agent according to the present invention, a reactive
15 organic compound having a reactive group may be used as the organic compound having the adhesiveness described above.

 With the present invention as described above, even when fiber having been subjected to the processing with the agent according to the present invention is used for a long period of time, it is possible to prevent the eggshell membrane from being removed.

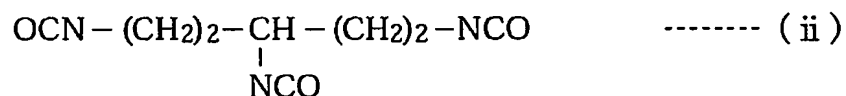
- 20 The fiber processing agent according to the present invention preferably contains one or more selected from the group consisting of (component 1) a hydrophilic compound having a polymerizable vinyl group in the molecule, (component 2) a monomer containing any of a hydroxyl group, a carboxylic group, an amino group, a sulfonic group, and a phosphate group, (component 3) a hydrophilic compound having an epoxy group,
25 (component 4) a compound having an aziridine group, and (component 5) a compound having an isocyanate group or a precursor thereof.

 Examples of the (component 1) to (component 4) contained in the reactive organic compound are the same as the (component 1) to (component 4) described as those contained in the fiber processing agent according to the present invention above, so that

descriptions thereof are omitted herefrom.

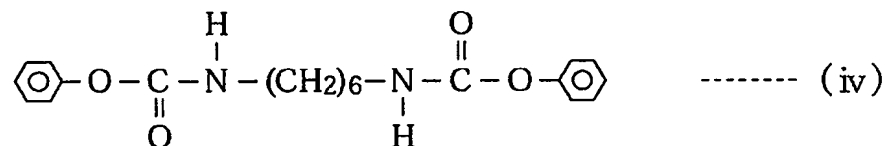
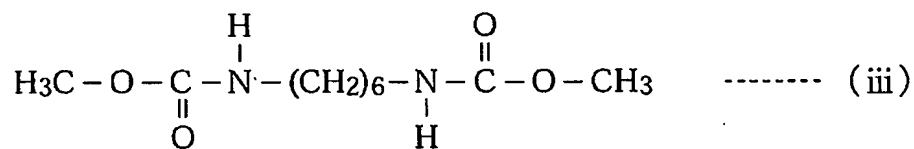
Examples of the (component 5) containing an isocyanate group are, for instance, hexamethylene diisocyanate and 1,3,5-trisocyanate-n-pentane expressed by the chemical formulas (i) and (ii) respectively:

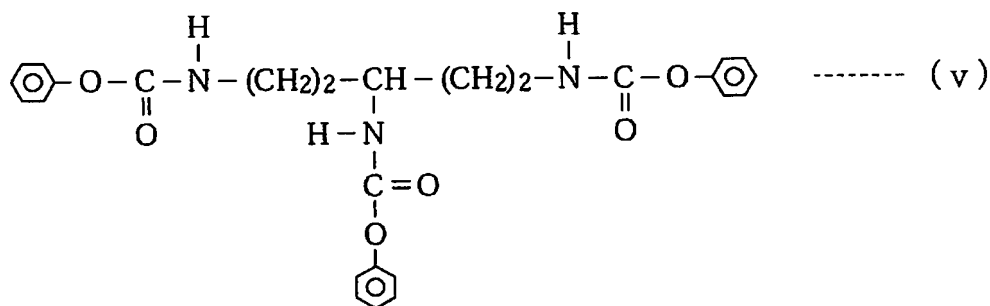
5



Examples of the (component 5) containing a precursor of an isocyanate group are, for instance, a precursor of hexamethylene diisocyanate and a precursor of 1,3,5-trisocyanate-n-pentane. Of these compounds, the precursor of hexamethylene diisocyanate are, for instance, 1, 6-di(methylcarbamoyl)-n-hexane and 1,6-di(phenylcarbamoyl)-n-hexane expressed by the chemical formulas (iii) and (iv) respectively. The precursor of 1,3,5-trisocyanate-n-pentane is, for instance, the compound expressed by the chemical formula (v) below:

15





In the fiber processing agent according to the present invention, as the organic compound having the adhesiveness as described above, any of lipophilic compounds such as wax or silicone and/or high molecular compounds such as ether, acryl, urethane, and those having an amide or ester group and the like may be used.

The organic compounds having the adhesiveness are those not reactive but having an appropriate degree of lipophilicity or hydrophilicity and adhere to a surface of fiber, and are used as a softening agent, a smoothing agent, an antistatic agent and the like.

More specifically, the lipophilic compounds are, for instance, wax, silicone, neutral fat, mineral oil, and animal wax. The high molecule compounds are, for instance, polyethylene glycol (di)alkylate, polyvalent alcohol ester, polyalkyl amide, polyacrylate.

With the invention as described above, the lipophilic compounds and/or high molecular compounds adhere to a surface of fiber, and elution of the eggshell membrane protein contained in the fiber to outside of the fiber can be prevented. Because of the characteristics, removal of the eggshell membrane can be prevented even when the fiber having been subjected to processing with the agent is used for a long period of time.

The fiber according to the present invention is characterized that the fiber is subjected to the fiber processing agent according to the present invention.

The fibers, which can be processed with the fiber processing agent according to the present invention, are, for instance, synthetic fibers such as nylon, polyester, acryl, and polyurethane; natural fibers such as cotton, linen, and wool; and compound fibers thereof.

Any method may be employed for processing with the fiber processing agent, and for instance, such methods as the immersing method, and padding method may be

employed. The immersing method includes, for instance, the room-temperature static method, and heating and agitating method.

The padding method includes, for instance, the pad dry method, and pad steam method. Any methods listed above may be employed in a case of the reactive organic
5 compound. In a case of a not-reactive compound, it is preferable to employ the pad dry method.

With the present invention as described above, as fibers are subjected to processing with the fiber processing agent, the fibers have excellent durability and can continuously show the moisture retention property, water-absorbing property,
10 moisture-absorbing property, antistatic property, and wound-treating property.

Best mode for Carrying out the Invention

The present invention is described more specifically with reference to examples and comparative examples.

15

[Example 1]

To prepare the fiber processing agent, at first an aqueous solution of the soluble eggshell membranes was prepared. Hen's eggs each with a shell was broken to remove the egg mixture, and the obtained eggshells each with an eggshell membrane were put in
20 clear water to manually remove the eggshell, and then the eggshell membrane were immersed for one hour in a 1% hydrochloric acid aqueous solution to dissolve fine eggshell pieces deposited on the eggshell membranes, and then the eggshell membranes were washed with water and naturally dried to obtain the eggshell membranes.

Then 1.5 liters of 5.0 mol/liter thioglycolic acid aqueous solution was added in
25 50 grams of the eggshell membranes obtained as described above. The thioglycolic acid aqueous solution containing the eggshell membranes was heated for 12 hours at a temperature of 60 °C to dissolve the eggshell membranes.

The aqueous solution with eggshell membranes dissolved therein was dialyzed through water acidified with hydrochloric acid to remove thioglycolic acid, thus the

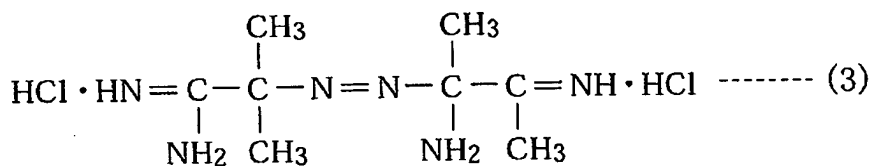
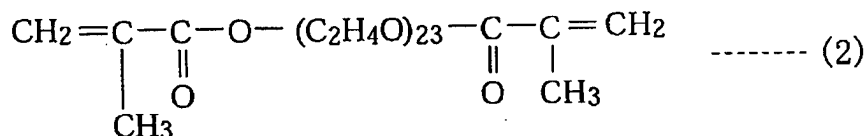
aqueous solution of the soluble eggshell membranes being obtained.

The aqueous solution of the soluble eggshell membrane and a reactive organic compound were blended with each other to prepare the fiber processing agent. Concentration for components in the fiber processing agent are as shown below:

5

Solid phase of the soluble eggshell membrane	5.0% by weight
Water	89.5% by weight
Reactive organic compound (expressed by the following chemical formula (2))	5.0% by weight

10	Reactive organic compound (expressed by the following chemical formula (3))	0.5% by weight
----	-----------------------------------------------------------------------------	----------------



15

The fiber processing agent prepared as described above was impregnated in the taffeta cloth containing 100% polyester (with the apparent specific gravity of 120 g/m²), and the cloth was wrung with a mangle to the agent content of 70%. Then the cloth was heated with steam for 10 minutes at a temperature of 105 °C, and then washed with hot water (for 10 minutes at a temperature of 40 °C), dried and thermally set.

20

[Example 2]

The taffeta cloth obtained according to the same procedure as that in Example 1 was washed with an automatic washing machine 10 times, and in each washing cycle the cloth was washed once and rinsed twice for 15 minutes.

25

[Example 3]

The aqueous solution of soluble eggshell membrane was blended with the Light Silicone PS-1000 (produced by KOEISHA CHEMCIAL Co. Ltd.) which is an organic compound with the adhesiveness to prepare the fiber processing agent according to a second aspect of the present invention. The aqueous solution of soluble eggshell membrane used for preparing the fiber processing agent was prepared according to the same procedure as that described in Example 1.

10	Solid phase of the soluble eggshell membrane	10.0% by weight
	Light Silicone PS-1000	10.0% by weight
	Water	80.0% by weight

Then the fiber processing agent prepared as described above was impregnated in the taffeta cloth containing 100% polyester (with the apparent specific gravity of 120 g/m²), and the cloth was wrung with a mangle to the agent content of 70%. Then the cloth was dried within a hot air drier for 5 minutes at a temperature of 150 °C, and then washed with an automatic washing machine in the same way as that employed in Example 2, namely 10 times and in each washing cycle the cloth was washed once and rinsed twice for 15 minutes.

[Comparative Example 1]

The same taffeta cloth containing only polyester as that employed in Example 1 was used, but the cloth was not subjected to the processing with the fiber processing agent, not to the other operations as described above.

[Comparative Example 2]

The fiber processing agent employed in this comparative example was different from that employed in Example 1 only in the point that the fiber processing agent

12

contained only the soluble eggshell membrane. Concentrations of the components of the fiber processing agent are as shown below:

	Solid phase of the soluble eggshell membrane	5% by weight
5	Water	95% by weight

The cloth was washed 10 times like in Example 2.

[Comparative Example 3]

- 10 The taffeta cloth was prepared with the fiber processing agent prepared by blending the components as shown below and according to the same procedure as that described in Example 3.

	Solid phase of the soluble eggshell membrane	10.0% by weight
	Water	90.0% by weight

15

Then the obtained taffeta cloth was washed 10 times line like in Example 3.

[Reference Example 1]

- 20 The taffeta cloth was prepared with the fiber processing agent prepared by blending the components as shown below and according to the same procedure as that described in Reference Example 3.

	Light Silicone PS-100	10.0% by weight
	Water	90.0% by weight

- 25 Then the obtained taffeta cloth was washed 10 times line like in Example 3.

[Assessment Method 1]

For the taffeta cloths according to the first aspect of the present invention in Examples 1, 2 and in Comparative Examples 1, 2, the moisture-absorbing property, a

water-absorbing rate, and friction-charged electrostatic potential were measured. Further for the taffeta cloths according to the second aspect of the present invention in Example 3, Comparative Example 3, and Reference Example 1, the water-absorbing rate and friction-charged electrostatic potential were measured. Measurement of the moisture-absorbing property was performed by placing a sample of the processed taffeta cloth for 12 hours in the atmospheric air with the relative humidity of 30% at a temperature of 23 °C for moisture control and measuring a change in the weight under the atmospheric air with the relative humidity of 80% at the temperature of 30 °C. The water-absorbing rate was measured by the JIS L 1096-A method. Further the friction-charged electrostatic potential was measured by the JIS L 1094-B method. A result of the assessment is shown in Table 1.

(Table 1)

	Moisture Absorbing property	Water Absorbing rate	Friction-charged electrostatic potential [V]
Example 1	2.1	1 sec. or below	200
Example 2	2.0	1 sec. or below	400
Com. Example 1	0.1	No absorption in 5 minutes or more	4800
Com. Example 2	0.2	No absorption in 5 minutes or more	4500
Example 3	-	1 to 5 sec	1400
Com. Example 3	-	No absorption in 5 minutes or more	4200
Ref. Example 1	-	2 to 5 minutes	2800

From the result of the Assessment Method 1 above, it is understood that the moisture-absorbing property and water-absorbing rate in Example 1 are higher as compared to those in Comparative Example 1 and the friction-charged electrostatic potential in Example 1 is smaller as compared to that in Comparative Example, which

indicates that Example 1 is superior to Comparative Example 1 in all aspects. From this result, it is understood that the fiber processed with the fiber processing agent according to the present invention (containing a reactive organic compound) was improved in the water-absorbing property, moisture-absorbing property, and antistatic property.

5 Further Example 2 shows the higher moisture-absorbing property and water-absorbing rate and smaller friction-charged electrostatic potential as compared to Comparative Example 2, which suggests that Example 2 is superior to Comparative Example 2 in all aspects. Therefore it is understood that the fiber processed with the fiber processing agent according to the present invention (containing a reactive organic
10 compound) does not lose the effects provided by the fiber processing agent even after washed repeatedly and has excellent durability.

In Comparative Example 2, the moisture-absorbing property is 20%, the water-absorbing rate is not more than one second, and the friction-charged electrostatic potential is 500 V in the initial state before washing. Comparison of the initial state to
15 the state after washing suggests that the fiber processing agent containing only the soluble eggshell membrane like that in the conventional technology does not improve the durability.

Comparing Example 3 to Comparative Example 3, it is understood that Example 3 shows the higher water-absorbing rate and smaller friction-charged electrostatic
20 potential as compared to Comparative Example 3, and that Example 3 is superior to Comparative Example 3 in all aspects. Also comparison of Example 3 to Reference Example 1 suggests the same conclusion. For the reasons as described above, it is understood that the fiber processed with the fiber processing agent according to the present invention (containing an organic compound having the adhesiveness) is improved
25 in the water-absorbing property and the antistatic property.

[Assessment Method 2]

After a commercially available adhesive tape is adhered on and peeled off from a human skin and then rough surface is intentionally produced with an acetone/ether

mixture solution, the taffeta cloths prepared in Examples and Comparative Examples were adhered and fixed on an upper arm for a specified number of days and for 6 hours each day, and conductance of the upper arm with the cloths wound around was measured. A result of the assessment is as shown in Table 2.

5

(Table 2)

		Day 0	Day 1	Day 3	Day 7	Day 14	Day 21
Conduc- tance[μ S]	Exam. 1	1.8	2.3	2.5	7.5	40.0	43.0
	C. E. 1	2.0	2.1	2.0	3.0	15.0	20.0

From a result of assessment according to the Assessment Method 2, it is understood that the conductance in Example 1 is larger as compared to that in Comparative Example 1. A larger value of this conductance indicates improved conductivity of human skin, which indicates that the roughed skin is being cured and the moisture retention of the skin is being improved.

For the reasons as described above, when the fiber processed with the fiber processing agent according to the present invention (containing a reactive organic compound) is used, a human skin having rough surface portions is cured, which indicates that the wound-treating property and moisture retention are improved. Even when used for a long period of time like in experiments according to this Assessment Method 2, the effects of wound-treating property and moisture retention are recognized, and therefore it is understood that the excellent properties are maintained from a long period of time.

20

Industrial Availability

The present invention provides a fiber processing agent capable of improving the various properties such as moisture retention, water-absorbing property, moisture-absorbing property, and antistatic property, and also provides fibers and fiber products improved in the properties as described above.

25

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.